

X-ray microscopy at electrochemical systems for sustainable energy technologies

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The development of new materials for efficient and durable systems used for sustainable energy technologies is crucial for current and future energy technologies. Performance and lifetime of these electrochemical systems depend on the 3D morphology of the materials and on morphology changes during operation. A profound understanding of degradation mechanisms that affect the performance and limit the lifetime of systems for energy storage and conversion is mandatory for more stable and robust solutions.

High-resolution X-ray imaging provides nondestructive characterization capabilities on opaque objects, observing features with sizes across a range of length scales, down to several 10 nanometers using lens-based full-field X-ray microscopy (TXM). Multi-scale X-ray computed tomography (XCT), characterized by a sample thickness / resolution value of $\sim 10^3$, and subsequent 3D data reconstruction is an efficient approach to study the 3D morphology of hierarchically structured systems and materials. Laboratory TXM and nano-XCT provide high-resolution 3D information about the morphology of materials for battery electrodes and electrocatalytic systems for water splitting. In an in-situ study, the 3D morphology of $\text{Na}_{0.9}\text{Fe}_{0.45}\text{Ti}_{1.55}\text{O}_4$ sodium iron titanate cathode material in Li-ion batteries and microcracks are imaged with sub-100nm resolution. The size- and density-dependence of the fracture behavior of the cathode-material particles is revealed based on a semi-quantitative analysis of the formation and propagation of microcracks in particles [1]. Multi-scale X-ray imaging of a novel transition-metal-based electrocatalytic system for water splitting provides 3D information of the morphology of the hierarchical $\text{MoNi}_4/\text{MoO}_2@/\text{Ni}$ structure - MoNi_4 electrocatalysts anchored on MoO_2 cuboids aligned on Ni foam -, a material system with high electrocatalytic efficiency for hydrogen evolution reaction. Micro-XCT images clearly resolve the Ni foam and the attached needle-like MoO_2 micro cuboids. Laboratory nano-XCT shows that the MoO_2 micro cuboids are vertically arranged on Ni foam. MoNi_4 nanoparticles with a size of 20 to 100 nm, positioned on single MoO_2 cuboids, were imaged using SR nano-XCT [2].

[1] V Shapovalov et al., *Crystals* 12 (1), 3 (2022);

[2] E. Zschech et al., *Micron* 158, 103262 (2022)